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Microbeam analysis — Electron backscatter diffraction — Measurement of average grain size

Analyse par microfaisceaux — Diffraction d'électrons rétrodiffusés — Mesurage de la taille moyenne des grains

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 202, *Microbeam analysis*.

This second edition cancels and replaces the first edition (ISO 13067:2011), which has been technically revised. The main changes compared to the previous edition are as follows:

— Data from a round robin (Annex B) have been used to:

  — Include information on expected precision (Clause 7 and Annex B);

  — Include more detail on sources of errors (Clause 7);

  — Clarify statements on minimum numbers of grains measured (5.8) and acceptable clean up procedures (6.3–6.3);

  — Clarify the distinction between sectional grain size measured on a 2D section and average grain size determined from some 2D measurements of grain sections which can be related by stereology to the 3D grain size;

  — Additionally, improvements have been made to the description of calculation of average values (6.5) and representation of the data (6.6).

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

The mechanical and electromagnetic properties of engineering materials are strongly influenced by their crystal grain size and distribution. For example, strength, toughness and hardness are all important engineering properties that are strongly influenced by these parameters. Both bulk materials and thin films, even as narrow two-dimensional structures, are influenced by grain size. For this reason, it is important to have standard methods for its measurement with commonly used and agreed terminology. This document describes procedures for measuring average grain size from maps of local orientation measurements using electron backscatter diffraction.

Microbeam analysis — Electron backscatter diffraction — Measurement of average grain size

1 Scope

This document describes procedures for measuring average grain size derived from a two-dimensional polished cross-section using electron backscatter diffraction (EBSD). This requires the measurement of orientation, misorientation and pattern quality factor as a function of position in the crystalline specimen[1]. The measurements in this document are made on two dimensional sections. The reader should note carefully the definitions used (3.3) which draw a distinction between the measured sectional grain sizes, and the mean grain size which can be derived from them that relates to the three dimensional grain size.

NOTE 1 While conventional methods for grain size determination using optical microscopy are well-established, EBSD methods offer a number of advantages over these techniques, including increased spatial resolution and quantitative description of the orientation of the grains.

NOTE 2 The method also lends itself to the measurement of the grain size of complex materials, for example those with a significant duplex content.

NOTE 3 The reader is warned to interpret the results with care when attempting to investigate specimens with high levels of deformation.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 16700, *Microbeam analysis — Scanning electron microscopy — Guidelines for calibrating image magnification*

ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories*

ISO 23833, *Microbeam analysis — Electron probe microanalysis (EPMA) — Vocabulary*

ISO 24173, *Microbeam analysis — Guidelines for orientation measurement using electron backscatter diffraction*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 24173 and ISO 23833 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1 Terminology associated with EBSD measurement of grain size

3.1.1
step size
distance between adjacent points from which individual EBSD patterns are acquired during collection of data for an EBSD map

3.1.2
pixel
picture element
smallest area of an EBSD map, with the dimensions of the *step size* (3.1.1), to which is assigned the result of a single *orientation* (3.1.3) measurement made by stopping the beam at a point at the centre of that area

3.1.3
orientation
mathematical description of the angular relationship between the crystal axes of the analysis point and a reference frame, usually the specimen axes

[SOURCE: ISO 24173:2009, 3.16, modified to include different reference frames.]

3.1.4
indexed
meets the predetermined threshold for reliability for the *orientation* (3.1.3) of a *pixel* (3.1.2) calculated from the EBSD pattern acquired for that pixel

3.1.5
indexing reliability
numerical value that indicates the confidence/reliability that the indexing software places in an automatic analysis

Note 1 to entry: This parameter varies between EBSD manufacturers, but can include:

a) the average difference between the experimentally determined angles between diffracting planes and those angles calculated for the orientation determined by EBSD software;

b) the difference between the number of triplets (intersections of three Kikuchi bands) in the EBSD pattern matched by the chosen orientation and the next best possible solution, divided by the total number of triplets.

3.1.6
orientation map
crystal orientation map
map-like display of *pixels* (3.1.2) derived from the sequential measurement of crystal *orientation* (3.1.3) at each point in a grid [see Figures 1 b) to 1 f)] showing the crystallographic relationship between the pixels and the reference frame

[SOURCE: ISO 24173:2009, 3.17, modified to include reference to examples.]

3.1.7
pattern quality
measure of the sharpness of the diffraction bands or the range of contrast within a diffraction pattern

Note 1 to entry: Different terms are used in different commercial software packages, including, for example, band contrast, band slope and image quality.

3.1.8
pattern quality map
map-like display of *pixels* (3.1.2) derived from the sequential collection of EBSD patterns at each point in a grid [see Figure 1 a)] showing the *pattern quality* (3.1.7) of the individual pixels

Note 1 to entry: Since measures of pattern quality can change at features such as grain boundaries and with orientation, the pattern quality map can give an indication of grain shape and size.

Note 2 to entry: Pattern quality maps can also indicate areas of heavy deformation and inadequate preparation, such as residual scratches.

Note 3 to entry: Small particles and features also contribute to the pattern quality map.

3.1.9
pseudosymmetry
potential for an EBSD pattern to be indexed in several different ways due to internal similarities within the EBSD pattern

Note 1 to entry: Pseudosymmetry is a problem with some crystal orientations, usually when a main zone axis is in the centre of the pattern. Typical cases are a {0001} pole for a hexagonal structure and a <111> pole for a cubic structure.

Note 2 to entry: Structures such as high-symmetry tetragonal crystals with an axial ratio, c/a, αpproximately equal to 1 are also likely to exhibit pseudosymmetry in EBSD patterns.

[SOURCE: ISO 24173:2009, 3.22]

3.1.10
misorientation
rotation, often defined by an angle/axis pair, required to rotate one set of crystal axes into coincidence with the other set of crystal axes, given two crystal o*rientations* (3.1.3)

3.1.11
disorientation
due to crystal symmetry, there can be several axis/angle pairs which represent the same misorientation, in which case the one having the smallest angle is called the disorientation

Note 1 to entry: For most crystal symmetries, there are multiple symmetrically equivalent axes for the disorientation with the smallest misorientation angle.

Note 2 to entry: Misorientation and disorientation are terms which are often used interchangeably. Disorientation is the more rigorous term here, but misorientation is the more frequently used.

3.1.12
forescatter imaging
orientation contrast produced from electrons which channel out of the specimen

Note 1 to entry: Other contrast mechanisms such as composition can also affect the contrast obtained.

3.1.13
electron-channelling contrast imaging
ECCI
orientation contrast produced from electrons which channel into the specimen

3.1.14
barrel distortion
difference in lateral magnification between the central and peripheral areas of an image such that the lateral magnification is less at the periphery

Note 1 to entry: A square object in the centre of the field appears barrel-shaped (i.e. with convex sides).

[SOURCE: ISO 10934-1:2002, 2.4.5.1]

3.1.15
pincushion distortion
difference in lateral magnification between the central and peripheral areas of an image such that the lateral magnification is greater at the periphery

Note 1 to entry: A square object in the centre of the field appears cushion-shaped (i.e. with concave edges).

[SOURCE: ISO 10934-1:2002, 2.4.5.2]

3.2 Terminology associated with grains and grain boundaries determined via EBSD

3.2.1
grain boundary
line separating adjacent regions of points in an EBSD orientation map with *disorientation* (3.1.11) across the line greater than a minimum angle chosen to define the grain boundaries

3.2.2
grain
region of points with similar *orientation* (3.1.3) (within a tolerance), completely enclosed by *grain boundaries* (3.2.1) and greater than the minimum size defined to exclude isolated (often badly *indexed* (3.1.4)) points as small grains

3.2.3
sub-grain boundary
line separating adjacent regions of points in a *grain* (3.2.2) with a difference in *orientation* (3.1.2) across the line smaller than that defining a *grain* (3.2.2) but greater than that defining a *sub-grain* (3.2.4)

Note 1 to entry: Effectively, sub-grain boundaries are grain boundaries with a smaller misorientation limit than that defining a grain boundary. These boundaries can have a characteristic linear appearance and exhibit a characteristic misorientation.

3.2.4
sub-grain
region of points with similar orientation completely enclosed by boundaries greater than the minimum *sub-grain boundary* (3.2.3) angle

3.2.5
special boundary
boundary between two *grains* (3.2.2) having a special *orientation* (3.1.3) relationship within a tolerance associated with identifying them in *orientation maps* (3.1.6)

3.2.6
twin boundary
particular case of a *special boundary* (3.2.5) between crystals oriented with respect to one another according to some symmetry rule, in which the boundary itself is planar and is a characteristic crystallographic plane (for both crystals) and, frequently, one crystal is the mirror image of the other

Note 1 to entry: For example, in face-centred-cubic structures, the characteristic misorientation defining a common twin can be described as a 60° rotation about the <111> axis with the boundary plane normal to the rotation axis.

3.2.7
recrystallized grains
new set of undeformed *grains* (3.2.2) formed by consuming deformed grains through nucleation and growth processes

Note 1 to entry: Measurements of misorientation within grains by EBSD can be used to distinguish between deformed and undeformed grains.

3.2.8
phase
physically homogeneous volume in a material having the same crystal structure and chemical composition

3.3 Terminology associated within grain size measurement

There are a variety of ways of representing average grain size. This subclause outlines some of the more common terms used, and the reader is referred to Annex A for more details about other terms, about the standards available and about the applicability of methods for particular grain shapes and distributions.

3.3.1
3D grain size
three-dimensional size of a *grain* (3.2.2) or crystal within a polycrystalline material, measured as a volume

Note 1 to entry: In a strict stereological definition, just the term grain size is sufficient to denote this value, but it is recommended to use the full description 3D grain size to avoid confusion with the *sectional grain size* (3.3.8) which is often shortened to grain size as well.

3.3.2
average grain size
value determined from a two dimensional measurement which is related to the average three dimensional size of a collection of grains or crystals forming a polycrystalline material by stereological relationships[2]. It can be reported as one or more of the following measurements:

a) average area

b) average diameter determined from average area

c) average linear intercept length

3.3.3
line intercept
distance between the points at which a straight line crossing a grain intersects the *grain boundary* (3.2.1) on each side

Note 1 to entry: See ASTM E112[15] for more details.

3.3.4
equivalent circle diameter
D_{circle}
diameter of the circle with an area equivalent to the grain section area, given by:

$$D_{\text{circle}} = (4A/\pi)^{1/2}$$

where A is the area of the *grain* (3.2.2)

Note 1 to entry: The ASTM grain size number, G, is given by[15]:

$$G = -6{,}64\log_{10}D_{\text{circle}} - 2{,}95$$

where D_{circle} is measured in mm.

3.3.5
Feret diameter
perpendicular distance between two parallel lines drawn in a given direction tangential to the perimeter of an object on opposite sides of the object

Note 1 to entry: It is also known as the calliper diameter.

Note 2 to entry: Different variants of the Feret diameter are used. For example, the Feret diameter can be measured in the vertical and horizontal directions or in any two directions at right angles to each other.

3.3.6
grain shape
property whose value is determined by fitting an ellipse round the *grain* (3.2.2) and measuring the aspect ratio, i.e. the ratio of the length of minor axis to the length of the major axis

Note 1 to entry: It is sometimes referred to as grain elongation.

Note 2 to entry: The value lies in the range 0 to 1.

Note 3 to entry: There are several ways of fitting the ellipse round the grain, and different methods can result in small differences in the measured aspect ratio.

3.3.7
grain shape orientation
angle between the major axis of an ellipse fitted round the *grain* (3.2.2) and the horizontal direction, usually measured counter clockwise

3.3.8
sectional grain size
two-dimensional size of a planar cross section through a *grain* (3.2.2), reported as

a) the area of the cross section

b) a diameter (see *circle equivalent diameter* (3.3.4) or *Feret diameter* (3.3.5))

Note 1 to entry: This is often shortened to grain size in common parlance, but can lead to confusion with the *3D grain size* (3.3.1).

Note 2 to entry: This is equivalent to the term projected grain size.

3.4 Terminology associated with data correction and uncertainty of EBSD maps

3.4.1
misindexing
assigning an incorrect *orientation* (3.1.3) or *phase* (3.2.8) to the measured EBSD pattern

Note 1 to entry: This can occur for a number of reasons, e.g. pseudosymmetry effects, attempting to index a poor pattern or attempting to index a pattern from an unanticipated phase for which the indexing software is not configured.

3.4.2
non-indexing
non-assignment of an *orientation* (3.1.3) due to insufficient quality of the EBSD pattern

Note 1 to entry: This can occur for a variety of reasons, such as roughness of the specimen, dust on the specimen, overlapping patterns at the grain boundary, a poor-quality pattern due to the effects of strain, or the pattern is from an unanticipated phase.

3.4.3
data cleaning
process chosen to accommodate *non-indexed* (3.4.2) and *misindexed* (3.4.1) data within the map, using a given set of parameters, typically based on the characteristics (*orientation* (3.1.3), *phase* (3.2.8)) of a certain number of nearest neighbours

Note 1 to entry: A wide range of terms (not necessarily mathematically precise) is used in the various commercially available software packages for different data-cleaning operations, including noise reduction, extrapolation, dilation and erosion.

Note 2 to entry: See Figures 1 b) to 1 f).

4 Equipment for grain sizing by EBSD

4.1 Hardware requirements

The reader is referred to ISO 24173 for equipment needed to acquire electron backscatter patterns, index the patterns (determine the orientation) and either step the beam across the specimen surface or, less commonly, step the stage, keeping the beam stationary to acquire a map.

4.2 Software requirements

4.2.1 The software shall allow the orientation data (or other parameters, such as pattern quality derived from each diffraction pattern) to be displayed as a map.

4.2.2 The software shall correct misindexed pixels or fill in non-indexed pixels (see 6.2 and 6.3).

4.2.3 The software shall use orientation data to define the positions of boundaries in accordance with the criteria selected.

4.2.4 The software shall identify grains as regions of connected pixels from the set of boundary points and measure grain size parameters. Special treatment may be applied to grains that intercept the map edges, e.g. removal or weighting.

5 Acquiring the map for grain sizing by EBSD

5.1 Specimen preparation

In order to achieve a high degree of indexing of individual pixels (a high indexing hit rate), it is necessary to produce a surface finish which produces EBSD patterns of sufficient quality to be indexed reliably. The criteria used for indexing reliability shall be defined and reported by the user.

The surface preparation method adopted will be dependent on the material and also on its condition e.g. metallurgical heat treatment. The reader should refer to standard texts on polishing and etching and ISO 24173:2009, Annex B. Over-etching of grain boundaries should be avoided since it leads to increased numbers of non- and mis-indexed points and to low index reliability at the grain boundaries.

If necessary, the specimen may be coated with a thin conductive coating (such as carbon) to prevent charging and electron beam drift and thus avoid distortion of the image.

5.2 Defining specimen axes

If the specimen is known to be strongly textured, e.g. from thermomechanical processing, the axes of the specimen shall be identified prior to preparation for EBSD such that EBSD measurements can be related to these axes. These axes are usually related to the rolling direction (for metals), to a growth direction (e.g., in thin films) or to a principal applied stress.

5.3 Stage positioning and calibration

The procedures set out in ISO 24173 shall be followed. The specimen shall be fixed to the scanning electron microscope (SEM) stage in the desired orientation with the specimen axes relative to the stage axes and imaged at a working distance at which the SEM and EBSD image magnifications have been calibrated and at which the EBSD system itself has been calibrated to index diffraction patterns.

The purpose of this calibration is to check that there is no influence of distortion on the recorded patterns and to ensure that the tilt angle relative to the specimen is correct. Reference [4] discusses distortion round the edges.

The specimen tilt has a significant effect on the image magnification in the direction on the specimen surface normal to the tilt axis. Great care shall be taken to measure the tilt angle of the specimen surface accurately.

NOTE A 1° change in tilt angle at a tilt angle at 70° will cause a change of ~5 % in the size of the step used in the direction on the specimen surface normal to the tilt axis when collecting data for the map.

5.4 Linear calibration

Follow the recommendations of ISO 16700.

5.5 Preliminary examination

An initial examination of the specimen shall be made to identify an initial set of operating parameters needed to map the orientation of the specimen with an acceptable level of accuracy and within an acceptable period of time over an area sufficient to give data on a statistically significant number of grains.

The reader is referred to ISO 24173 for information needed to measure the orientation.

5.6 Choice of step size

5.6.1 If the grain size and shape are not known already, an approximate grain size and shape estimation shall be performed by a quick imaging technique. An optical microscope might work on a region with only slight polishing relief or on an etched region adjacent to that to be examined by EBSD. Forescatter[5] or electron-channelling contrast imaging using diodes mounted on the EBSD detector, or imaging with the specimen current, can also produce images relatively quickly.

As an alternative to mapping, some EBSD software offers a line intercept method as a mapping mode. This can be used to quickly give an approximate grain size measurement.

5.6.2 The step size should be chosen in relation to the average grain size, unless information on a particular minimum size is required. In either case, it shall be recognized that a judgement is being made on the minimum number of pixels that are used to define a grain either by a lineal or areal method. See also 6.3 and Figures 1 d), e) and f) for the effects of step size choice.

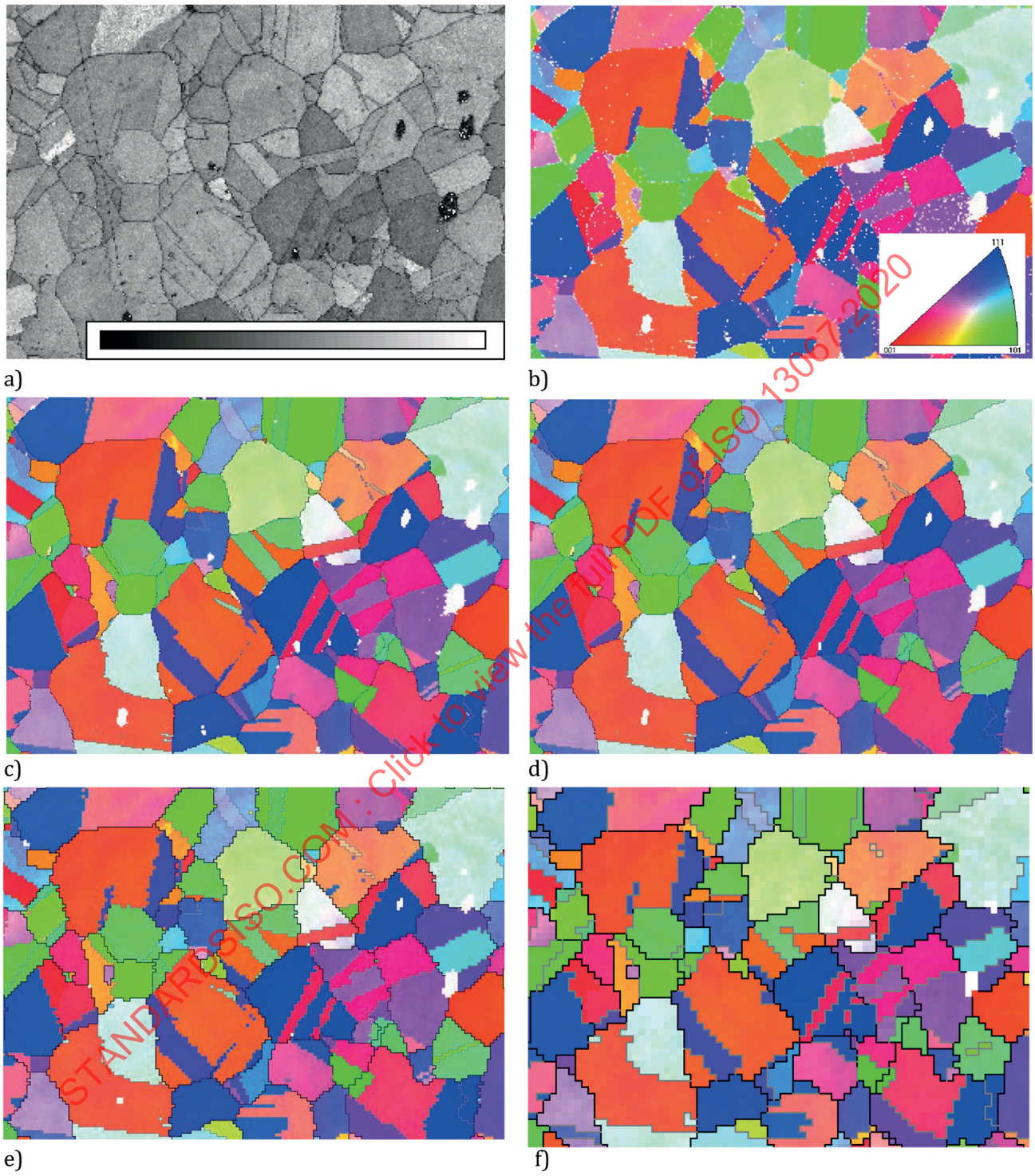


Figure 1 — An area of a Ni superalloy mapped by EBSD under different conditions

Figure 1 shows, using Ni as an example material to illustrate general principles:

a) a pattern quality map (grey-scale range covering 20 to 160 of 256 grey levels), generated using a 0,5 µm step size;

b) from the same data set, the raw orientation map (96,7 % indexed) with non-indexed points in white and inverse pole figure colouring of orientations (specimen normal direction, with key bottom right);

c) Figure 1 b) after removing clusters of 3 pixels or less and replacing the unindexed pixels by orientations based on their six nearest neighbours (99,3 % indexed);

d) similar to Figure 1 c), but based on two instead of six nearest neighbours (99,8 % indexed);

e) the same area mapped with a 1 µm step size;

f) the same area mapped with a 2 µm step size.

Figures 1 c) to f) all use the orientation key shown in Figure 1 b) and show grain boundaries (>10°) in black and twin boundaries (60° ± 1°, [111] ± 1°) in grey.

A simple rule that can be applied to a preliminary scan is that the step size should be less than 10 % of the approximate mean grain size[3]. Annex B summarises some effects of choice of step size determined by an interlaboratory comparison[6]. To confirm the validity of the chosen step size, repeat the mapping of a single area at several step sizes and determine the maximum size below which no significant difference in average grain size is determined. This choice has a direct influence on the accuracy of the grain size measurement.

5.6.3 In choosing the step size, the spatial resolution of the system needs to be considered. The step size is preferably larger than the interaction volume, which will be determined both by the material examined and the operating parameters of the SEM, such as the filament type, accelerating voltage and aperture size.

5.7 Determination of the level of angular accuracy needed[7][8]

The speed with which EBSD patterns are acquired (including any averaging of patterns) may affect the precision with which band edges can be detected and thus the angular accuracy of the calculated orientation. Other factors, such as the Hough resolution and the number of bands chosen to match the calculated orientation, also affect the calculation time as well as the angular accuracy.

If too long a time is taken for acquisition and calculation, problems of specimen drift can be increased significantly, and fewer points will be acquired in a given time, reducing the statistical significance of the data acquired. To minimize drift, it is recommended that the specimen have a good earth (ground) path and is securely fastened to the stage. Avoid carbon tabs. A thin carbon coating might also be necessary for insulating specimens.

If the time taken is too short, then levels of indexing reliability will be reduced. The settings chosen as a compromise between the two opposing factors above shall be recorded.

In some cases, saving of EBSD patterns without indexing during mapping may speed up map acquisition. Subsequent indexing off-line enables investigation of the effect of some of the above parameters on indexing accuracy. The approach of saving patterns for subsequent analysis is strongly recommended, not just as a way of speeding up data acquisition but also to allow flexibility in data interpretation.

5.8 Choice of areas to be mapped and map size

The areas chosen for examination shall be representative of the microstructure as a whole, and, if there is variation with position in the specimen, the positions examined shall be recorded in relation to the specimen geometry.

For conventional linear-intercept measurements, standards such as ASTM E112 recommend measurement of a minimum of 50 grains from a minimum of 3 fields. An EBSD interlaboratory study[6] (Annex B) demonstrated that measurement of 300 grains generally produces a stable, repeatable value on a running-average plot and that measurement of too few grains with a small step size can lead to significant errors by missing large grains. It is therefore recommended that a minimum of 300 grains per field is measured for a minimum of 3 fields, and that grains at the edge of each field shall be excluded from calculations.

If this minimum proves impossible (because for example of a very large grain size) then grains cutting the edge of a field may be included if the "bias" incurred by including partial grain areas can be compensated for by, for example, the Miles-Lantuéjoul correction[9] which assigns each particle a weight that is proportional to the chance it has of being contained within the measurement field.

With some equipment and software, it is possible to join together EBSD maps of adjacent areas. This should be avoided since joining maps in this way can lead to errors of alignment and the creation of false boundaries. Since grain size is a statistical quantity, it is better practice to take measurements on several separate areas.

NOTE 1 If statistical tools can be used to reduce errors in joining maps together, this process of grouping maps could be of interest.

NOTE 2 Difficulties in aligning images might be caused by using too low a magnification, giving rise to aberrations in the images, such as radial distortions (e.g. pincushion and barrel distortion) and scan rotation or an incorrectly set up SEM that shows poor orthogonality in the scan. Orthogonality errors can be observed and corrected for with the aid of a rectangular grid.

5.9 Considerations when examining plastically deformed materials

Where there is a high degree of damage, e.g. from plastic deformation, it might be impossible to obtain good diffraction patterns. This makes indexing impossible or leads to inaccurate measurement of orientation or phase. 6.2 and 6.3 consider the treatment of maps where this occurs, but it should be noted that, in cases where a substantial number (>10 %) of pixels are not indexed reliably, this treatment can distort the results and introduce significant inaccuracies.

Furthermore, deformation often leads to the formation of new grains and sub-grain boundaries. However, there is no universally agreed definition of the misorientation angles that define these boundaries since the significance of the boundary angle will vary depending to material type and the property under consideration. Thus, even if good indexing is achieved, it is essential that any measurement of size in a deformed material specify the misorientation angle used to define a grain boundary.

Heavily deformed microstructures can also show significant anisotropy, and several definitions might be needed to give representative descriptions of the grain size.

A further possible consequence of deformation, particularly at elevated temperatures, is the formation of strain-free recrystallized grains. In such cases, these grains might have a significantly larger grain size than the initial grains, resulting in a bimodal grain size distribution and the need to map at different step sizes to resolve the distribution.

6 Analytical procedure

6.1 Definition of boundaries

6.1.1 Grain boundary angles

After following the steps above and acquiring data to plot, for example, maps of orientation, grain boundaries can be drawn on the maps. This requires the angles defining the various possible boundaries to be chosen. Guidelines for this are given below but, whether these or other methods are used, the definitions and procedures used for grain size values shall be stated with all results.

For relatively simple equiaxed grain structures, such as fully recrystallized metals or undeformed cast metals, the misorientation that is used to define the grain boundary may be taken to be as little as 5°. For these types of material, there is evidence that misorientation angles between 5° and 15° make little difference to the average grain size[10].

For other materials, with more complicated grain structures, larger angles, typically 10° or 15°, depending on material, are used. Measurement of grain size as a function of misorientation angle might

be useful in gathering information on the structure. Care shall be taken to ensure that the prescribed angle is not so large that it results in two or more distinct, preferred orientations being encompassed within the angular range[11][12].

6.1.2 Handling incomplete boundaries

In some materials, particularly after deformation, selected boundaries might not extend completely between two regions to terminate at a triple point with another boundary because the measured misorientation changes along the length of the boundary fall below the defined grain boundary angle. In such cases, it might be possible to extrapolate the boundary by reducing the minimum angle generally used elsewhere in the map (see 5.6) to a new, lower, value. If this is done, it shall be recorded with the final result (preferably reporting the effect on mean size with and without extrapolation). It is, however, preferable to measure size with reduced misorientation angles defining grain boundaries and to note the effect of this reduction on the measured grain or sub-grain size.

6.1.3 Dealing with special boundaries

With conventional techniques, special boundaries such as twins in cubic materials, which can be identified by their morphology, are frequently ignored for the purposes of grain size measurement. Since EBSD measures angle/axis quantitatively, these boundaries can be easily determined by software and excluded from EBSD measurements of grain size. However, since there will be some variation in measured angle and axis about the idealized value, the tolerances used to define the boundaries shall be recorded (e.g. ±2° from 60° about <111>). The following should also be noted:

a) EBSD will define some boundaries as twins because they meet the defined misorientation tolerances, whereas conventional optical microscopy would not identify them because the typical morphology of twinning is not obvious. This effect can be reduced by only including those boundary segments with a trace, which also satisfies the requirements for a twin plane[13].

b) Removal of grain boundaries in a) will lead to larger grain sizes than if twins are included.

6.2 Post-acquisition treatment of raw data

Rarely will every pixel in an EBSD orientation map be correctly indexed. In addition to errors in orientation measurement for each pixel indexed, some pixels will not be indexed. The relative proportions of these pixels will depend on specimen preparation, the nature of the specimen, the SEM operating conditions and the EBSD indexing parameters.

In simple recrystallized specimens, it is normally possible to achieve a level of 95 % of pixels with acceptably high index reliability. This level of 95 % should be the target for all maps, but it was shown in an Interlaboratory study[6], (Annex B) that if misindexing is random then a minimum of 80 % with acceptably high index reliability (and following data-cleaning steps described in 6.3) will still produce acceptable results for mean grain size. However, incorrect or excessive manipulation of the raw data can alter the final measured sizes significantly.

NOTE In multiphase materials, it can be necessary to treat each phase differently, using a separate dataset for each phase.

6.3 Data-cleaning steps

6.3.1 Remove data for all grain sections with a number of pixels lower than a user-defined value (typically 3 to 5)[6]. See also 6.3.4 for removal of the smallest grain sections after image processing. The threshold value and the number removed shall be recorded.

6.3.2 Index any singly unindexed pixel where it is surrounded by x or more pixels of the same orientation, where the value of x is dependent on the grid used for mapping (square or hexagonal). Care needs to be taken if this process is repeated several times (a single pass is generally sufficient for non-indexed points at grain boundaries). The percentage indexed should not be increased by more than,

typically, 5 % but it has been shown[6] (Annex B) that, in the case of random misindexing, up to 10 % of pixels may be re-indexed. The percentage indexed in this manner shall be reported.

The effect of data cleaning (which has the potential to introduce artefacts) shall be investigated according to the information required on the grain size. (For example, the mean value might be less sensitive to data cleaning than the whole histogram, in particular for the smaller grain sizes.) The histogram of sectional grain size given by analysis of the raw data shall be recorded to assess any excessive artefacts introduced by data cleaning.

6.3.3 Optionally, an orientation filter, such as Kuwahara filter[14], may be used to reduce errors in orientation measurement. This is especially important for heavily deformed specimens. Use of such a filter shall be recorded with the results.

It should be borne in mind that the Kuwahara filter can introduce diagonal features into maps, and care should be taken to check that the features produced are actually present in the microstructure, e.g. by looking at the forescatter image or EBSP quality map.

6.3.4 Choose the minimum dimensions to be included in the calculations of the grain size. For conventional linear-intercept measurements, the minimum recommended length to measure is 10 pixels (so that in the worst case a +1 pixel error at one end and a –1 pixel error at the other end would give a maximum error of 20 %); this would suggest a minimum grain size area of 100 pixels. However, it has been shown[3][10] that measurements for all grain sections >10 pixels in area gives valid results since EBSD validates each pixel in a grain by measuring its orientation. Pixelation errors, which alter the true grain area at small sizes, are approximately 5 % for an area of 10 pixels.

It is therefore recommended that all grain sections >10 pixels in area should be included in the calculation of grain size. This agrees approximately with conventional image analysis, where the minimum size of an object is 9 (3 · 3) pixels for a square grid or 7 for a hexagonal grid to avoid erosion by a single layer of pixels deleting an object completely. A graph of average grain size against minimum size included in the calculation can be produced to confirm this choice of minimum and, if required, EBSD maps to highlight the grains excluded from the average grain size calculation can be produced.

If linear intercept measurements are made of grain size, then it is also recommended[6] that only intercept lengths of ≥10 pixels shall be included in the calculation of grain size to eliminate artefacts caused by lines running on and parallel to grain boundaries.

It is further recommended that, for equiaxed structures, intercept measurements are made in orthogonal directions parallel and perpendicular to the sample tilt axis. The calculation of an average size (see 6.5) for the two directions will give a good indication if sample or image movement (drift) during measurement was significant. If the average values differ by more than 10 % then repeat measurements should be made to investigate the cause.

For non-equiaxed structures then linear intercept measurements should be made in directions appropriate to the sample axes.

6.3.5 Maps showing (a) grains of more than 10 pixels and (b) grains of less than 10 pixels shall be investigated alongside maps of diffraction pattern quality which might highlight the significance of any data omitted from the calculation.

6.3.6 All grains touching the edge of a map shall be excluded from calculations of grain size (see also 5.8).

6.3.7 Figures 2 to 4 show examples of the effects of some of the above methods on the measurement of grain sizes in the nickel material used to make the maps shown in Figure 1.

Figure 2 shows cumulative sectional grain distributions from a larger region of the 0,5 µm step size maps shown in Figures 1 b) to d), resulting from various data-cleaning methods:

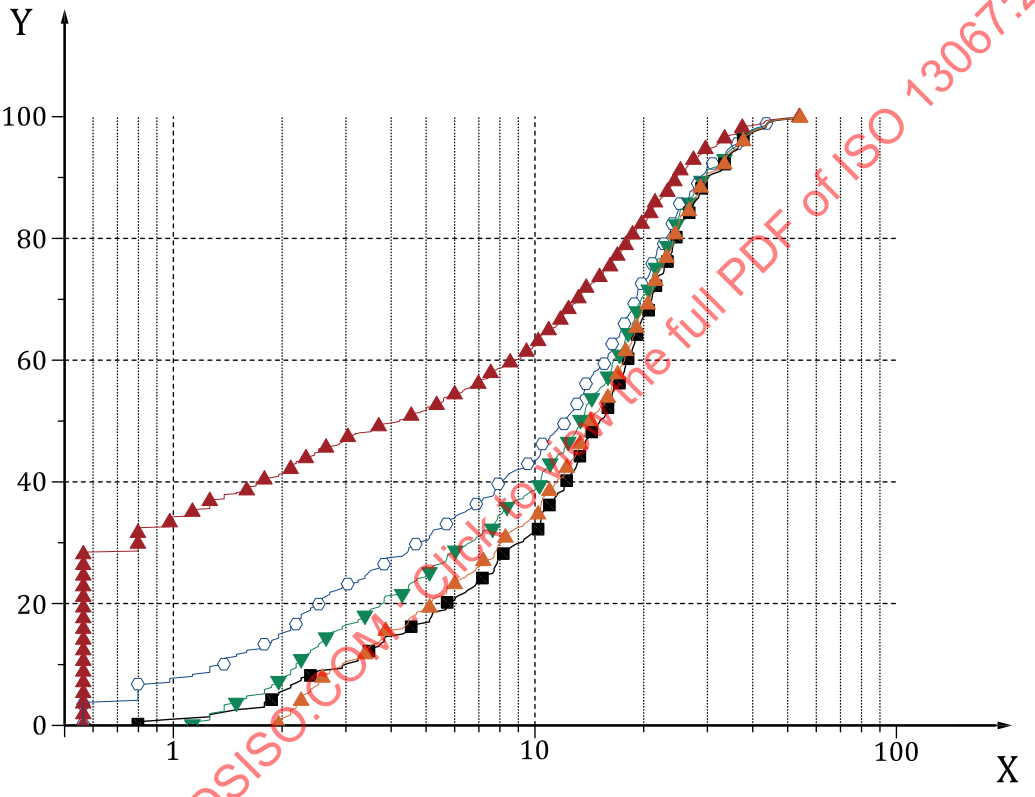
a) raw data;

b) removed single isolated pixels, dilated into unindexed pixels where five neighbours indexed;

c) as b), but removed 3-pixel clusters;

d) as b), but removed 10-pixel clusters;

e) as b), followed by removal of all grain sections with two or fewer neighbours.



Key

X sectional grain size (circle equivalent diameter), expressed in µm

Y cumulative probability, expressed in %

a)

b)

c)

d)

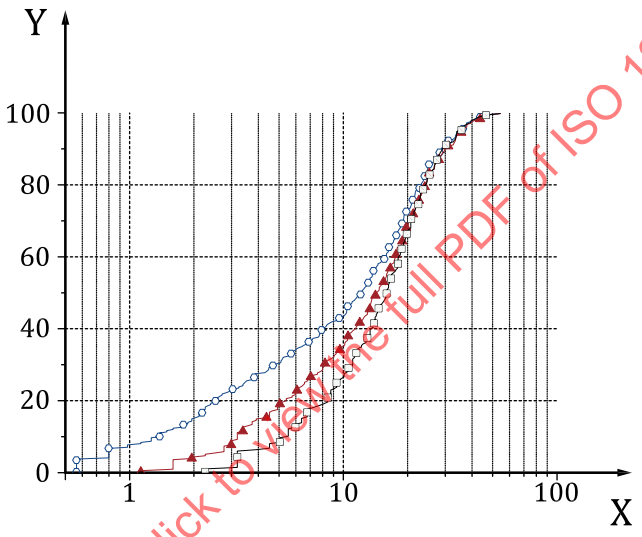
e)

Figure 2 — Cumulative sectional grain size distributions resulting from different data-cleaning methods

Figure 3 shows cumulative sectional grain size distributions resulting from maps of the same area produced with step sizes of a) 0,5 µm, b) 1 µm and c) 2 µm, [the 0,5 µm data is the same as that shown in Figure 2)] after data-cleaning steps involving the removal of single isolated pixels and dilation into unindexed pixels where five neighbours were indexed. See also Table 1.

Table 1 — Effect of pixel removal on mean grain size

Data-cleaning method		0,5 µm		1,0 µm		2,0 µm	
		Size µm	Number	Size µm	Number	Size µm	Number
a)	Raw data	9,3	571				
b)	Single pixels removed	13,7	304	15,7	265	16,9	242
c)	Three pixels removed	14,8	280				
d)	Ten pixels removed	15,7	261				
e)	Grains with <2 neighbouring grains removed	16,1	250	16,6	248	17,5	231



Key

X sectional grain size (circle equivalent diameter), expressed in µm

Y cumulative probability, expressed in %

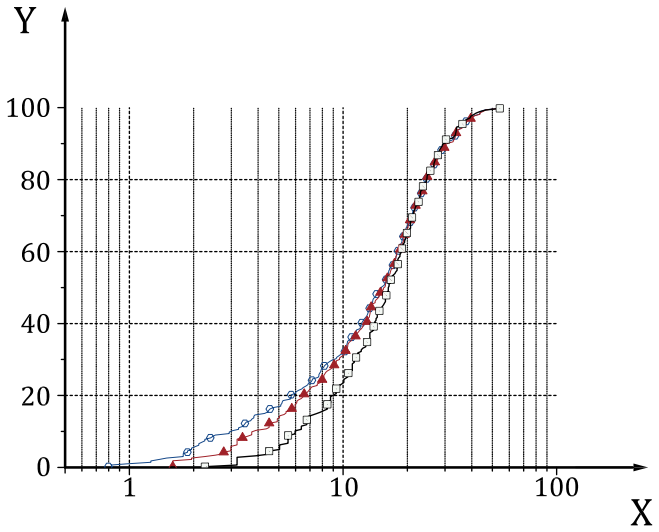
a)

b)

c)

Figure 3 — Cumulative sectional grain size distributions resulting from different map step sizes and single-pixel removal during data cleaning

Figure 4 shows cumulative sectional grain size distributions resulting from maps of the same area produced with step sizes of a) 0,5 µm, b) 1 µm and c) 2 µm, [the 0,5 µm data is the same as that shown in Figure 2)] after data-cleaning steps involving the removal of grains with two or fewer neighbours and dilation into unindexed pixels where five neighbours were indexed.



Key

X sectional grain size (circle equivalent diameter), expressed in µm

Y cumulative probability, expressed in %

a)

b)

c)

Figure 4 — Cumulative sectional grain size distributions resulting from different map step sizes and removal of grains with two or fewer neighbours during data cleaning

Following the recommendations in this document, only the results for the 0,5 µm and 1 µm step sizes (with a step size <0,1 times the average grain size) should be considered after rejecting features with a size of less than 10 pixels or after removing grains with fewer than two neighbours. The agreement between these methods is better than 6 %.

6.4 Measurement of sectional grain size

Sectional grain size can be measured in many ways, of which the most commonly reported are diameters based on linear intercept, circle equivalent diameter, feret diameters or area. Which of these is used depends on the application and whether comparison is being made with other existing measurement techniques.

6.5 Calculation of average grain size

For each EBSD orientation map an analysis of all grains identified after completing the procedures outlined in 6.1-6.3 will produce a list of areas of each individual grain section and therefore commonly individual D_{circle} values calculated from the areas (3.3.2). A list of individual linear intercept lengths may also be produced.

The different parameter lists present options for calculating an average grain size, including:

$$\overline{D_{\text{circle}(A)}} = \sqrt{\left(4\bar{A}/\pi\right)} \text{ or } \bar{A} = \frac{\sum A}{n}, \text{ or } \bar{l} = \frac{\sum l}{n}$$

But note that an alternative calculation $D_{\text{circle(n)}}$, which could be described as an average sectional grain size, is commonly used and will give a different value from $\overline{D_{\text{circle}(A)}}$:

$$\overline{D_{\text{circle}(n)}} = \frac{\sum D_{\text{circle}}}{n}$$

where

A is the grain area (in square µm);

D_{circle} is the equivalent circle diameter;

l is the intercept length (in µm);

n is the number of measurements of grain area or intercept lengths.

Area based measurements are often reported as a grain size index based on a count of the number of grains occupying a defined area and the value of $\bar{A}$ may be used to calculate an index number (see Annex A). If an index number is calculated, the reference number of any standard used in the derivation of this value shall be reported.

6.6 Representation of data

The average grain size for the sample analysed shall be calculated from the average values determined for each individual mapped area (6.5) and reported with the uncertainty quoted at the 95 % confidence level.

The sectional grain size distribution shall also be reported, including maximum, minimum and standard deviation and values for D10, D50 and D90. The size distribution shall be plotted as a cumulative distribution and may also be plotted as a binned histogram. Consideration should be given to whether to plot by size as a function of number or by size as a function of number weighted by area.

7 Measurement uncertainty

An estimate of the Type A uncertainties can be determined from the standard deviation between mean values calculated from the multiple maps (5.8) acquired from a given sample. Estimation of the relative effects of other uncertainties listed at the end of this section are difficult, but results of an interlaboratory study[6] on an equiaxed recrystallized Titanium sample which followed a draft version of this standard produced a measure of the precision of the technique given in Table 2 below.

Table 2 — Precision of the measurement of average sectional grain size of a Titanium alloy for equivalent circle diameter $\left(\overline{D_{\text{circle}(n)}} = \frac{\sum D_{\text{circle}}}{n}\right)$ and linear intercept measurement $\left(\bar{l} = \frac{\sum l}{n}\right)$

		Equivalent circle diameter (µm)	Linear intercept (µm)
Overall mean		33,5	30,1
Standard deviation of cell averages		1,88	1,02
Repeatability standard deviation	s_r	0,88	0,74
Reproducibility standard deviation	s_R	1,98	1,15
Repeatability limit	r	2,46	2,07
Reproducibility limit	R	5,55	3,22

The following factors should be noted from the report on the interlaboratory study[6]:

Major errors were caused by:

a) Calibration errors for magnification or scan orthogonality

b) Lack of data clean up

c) Too small a step size resulting in scanning an area that was too small and therefore not measuring large grains

Other factors which were found to contribute to uncertainty included:

a) The minimum grain size chosen to exclude from the calculation, which was in turn affected by

b) The choice of step size

These factors had a greater influence on the uncertainty than:

a) The level of accurate indexing of pixels above 80 %

b) The precise method used to clean up the maps for poorly indexed pixels

Other factors which may contribute to uncertainty but which were not quantified included:

a) Sample stability or drift in the area mapped by EBSD

b) Accuracy with which the tilt angle of the sample is known

8 Reporting of analysis results

8.1 The reporting of the analysis results shall be done in accordance with ISO/IEC 17025.

8.2 The specimen preparation method and section analysed shall be clearly indicated, together with the reference number of any standard used for metallographic evaluation of the grain size (see Annex A).

8.3 The SEM and EBSD operating conditions shall be indicated. Indicate gun type, accelerating voltage, working distance, probe current (if available), specimen tilt angle and type of scan (beam/stage), step size, magnification (or image width) and grid shape (square or hexagonal). State the phase and crystallographic structures used for EBSD indexing. The report shall also state the criteria used for reliable indexing and the methods of data cleaning used (see also 8.4).

8.4 If requested by the customer, a map showing the raw data (the data prior to any data-cleaning operations) shall be displayed, to allow the customer to check for artefacts due to the data-cleaning procedures. Report all data-cleaning operations, with the percentage of points cleaned, and the minimum grain size and the misorientation angle used to define the boundaries.

8.5 The data reported shall follow the requirements of 6.6.

Annex A
(informative)

Grain size measurement

The mechanical properties of engineering materials are strongly influenced by their crystal grain size and distribution. For example, strength, toughness and hardness are all important engineering properties that are strongly influenced by these parameters. It is therefore important to have standard methods, with commonly used and agreed terminology, for their measurement. Standardized methods of measurement of structure ensure that materials with repeatable properties are produced and the link between processing and structure reinforced.

Since materials exhibit a range of sectional grain sizes in a 2D cross section, the material can be characterised by both a size distribution and a mean value. Most existing and widely used standards for grain size measurement (such as ASTM E112[15], ASTM E930[16], ASTM E1181[17], ASTM E1382[18] and ISO 13383-1[21]) aim at measurement and reporting of a mean value, generally based on a measurement of the number of grains within an area, or on the number of grains intercepted by a line drawn across the structure. These therefore calculate an average grain size based on an average area, $\overline{A}$.

When a structure is mapped by EBSD, the data generated enables the automatic measurement of individual grain size areas or lengths, for all grains within the mapped area. The production of data on the distribution of sizes as well as the mean value is thus greatly simplified. The measurement by EBSD of phase and orientation at each pixel in a map also facilitates the clear definition of grain boundaries and identification of very small grains, based on the commonality of orientation in adjacent pixels. This identification of very small grains does however mean that there is scope for producing very different mean values if account is not taken of the small area fraction that they contribute and emphasises the need to examine grain size distribution as well as mean grain size.

Subclauses 6.4 to 6.6 of this document describe the options for reporting of mean grain size and *sectional* grain size distribution. If comparisons are to be made with mean sizes determined by other grain size standards then care must be taken to understand the differences both in method and calculation of grain size. In particular it should be noted that

$$\overline{D_{\text{circle}(n)}} = \frac{\Sigma D_{\text{circle}}}{n} \neq \overline{D_{\text{circle}}(A)} = \sqrt{\left(\frac{4\overline{A}}{\pi}\right)}$$

And therefore care must be taken when quoting the relationship:

$$\overline{D_{\text{circle}}(A)} = \sqrt{\left(\frac{4\overline{A}}{\pi}\right)} = \sqrt{\frac{4}{\pi}}\sqrt{\overline{A}} = 1{,}13\sqrt{\overline{A}} = 1{,}13\overline{l}$$

From the empirical relationship $\overline{l} = \sqrt{\overline{A}}$ quoted in Reference [19].

It should be noted that grain sizes given by D_{circle} will not be normally distributed and therefore the mean size determined from a simple average may not be the best representation of the distribution curve. Consideration may therefore be given to use of area weighted averages or use of other distributions such as log-normal and any average thus calculated

Other standards relevant to grain size measurement include:

Table A.1 — Other standards relevant to grain size measurement

ASTM E112	Grain size Heyn linear-intercept method Jeffries planimetric method
ASTM E1181	Duplex grain sizes
ASTM E1382	Grain size using image analysis
ASTM E930	Largest grain size
ISO 13383-1	Grain sizes in ceramics
ISO 643	Ferritic or Austenitic grain size in steels
ASTM E2627	Grain size by EBSD in Fully Recrystallized Polycrystalline Materials based on the approach of E112

Note that for measurement of duplex grain sizes, or of partially recrystallized materials EBSD measurements will produce a sectional grain size distribution which will clearly show the presence or otherwise of a duplex distribution or can be used to distinguish recrystallized from non-recrystallized grains based on parameters such as average grain misorientation.